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Lewis Research Center
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SUMMARY

An isothermal cross section of the tantalum-hafnium-carbon (Ta-Hf-C) system at 2050° C was constructed from x-ray diffraction, chemical analysis, metallographic, and microhardness data. No phases other than those appearing in the three binary systems were observed in the ternary system. A liquid phase was found in a large portion of the hafnium-rich corner of the ternary section.

INTRODUCTION

The melting points of tantalum carbide (TaC) and hafnium carbide (HfC) are very high (≈3950° and 3900° C, respectively) and the 4-tantalum-carbide - hafnium-carbide (4 TaC · HfC) solid solution is a material of the highest known melting point (≈4100° C)(ref. 1). Therefore, compositions within the TaC-HfC pseudobinary system are considered for possible application in the field of high-temperature thermionic energy conversion. In this connection vaporization studies have been conducted (ref. 2) in order to establish the thermal stability of TaC-HfC solid solutions. In the course of that investigation it was observed that compositional changes occurred on the surface of these carbides due to differences in the vaporization rates of tantalum (Ta), hafnium (Hf), and carbon (C). Consequently, a need became apparent for the knowledge of ternary equilibrium phase relations. An experimental study was initiated to determine the ternary diagram at 2050° C. This temperature was chosen because it is within the range of possible thermionic emitter operation. While the present work was in progress, an isothermal section of this system at 1850° C was reported (ref. 3).

Previous literature related to the present work includes binary diagrams for Ta-C (ref. 4), Hf-C (ref. 5), Ta-Hf (ref. 6), and ternary phase relations of Ta-Hf-C at 1850° C (ref. 3). These are shown in figure 1.

In the investigation reported here, the isothermal section at 2050° C of the Ta-Hf-C system was determined. Samples were prepared by several powder-metallurgical techniques and by arc melting. After homogenization heat treatment at 2050° C in vacuum, samples were rapidly cooled, and phase identification was made by x-ray diffraction, metallographic, and microhardness techniques.

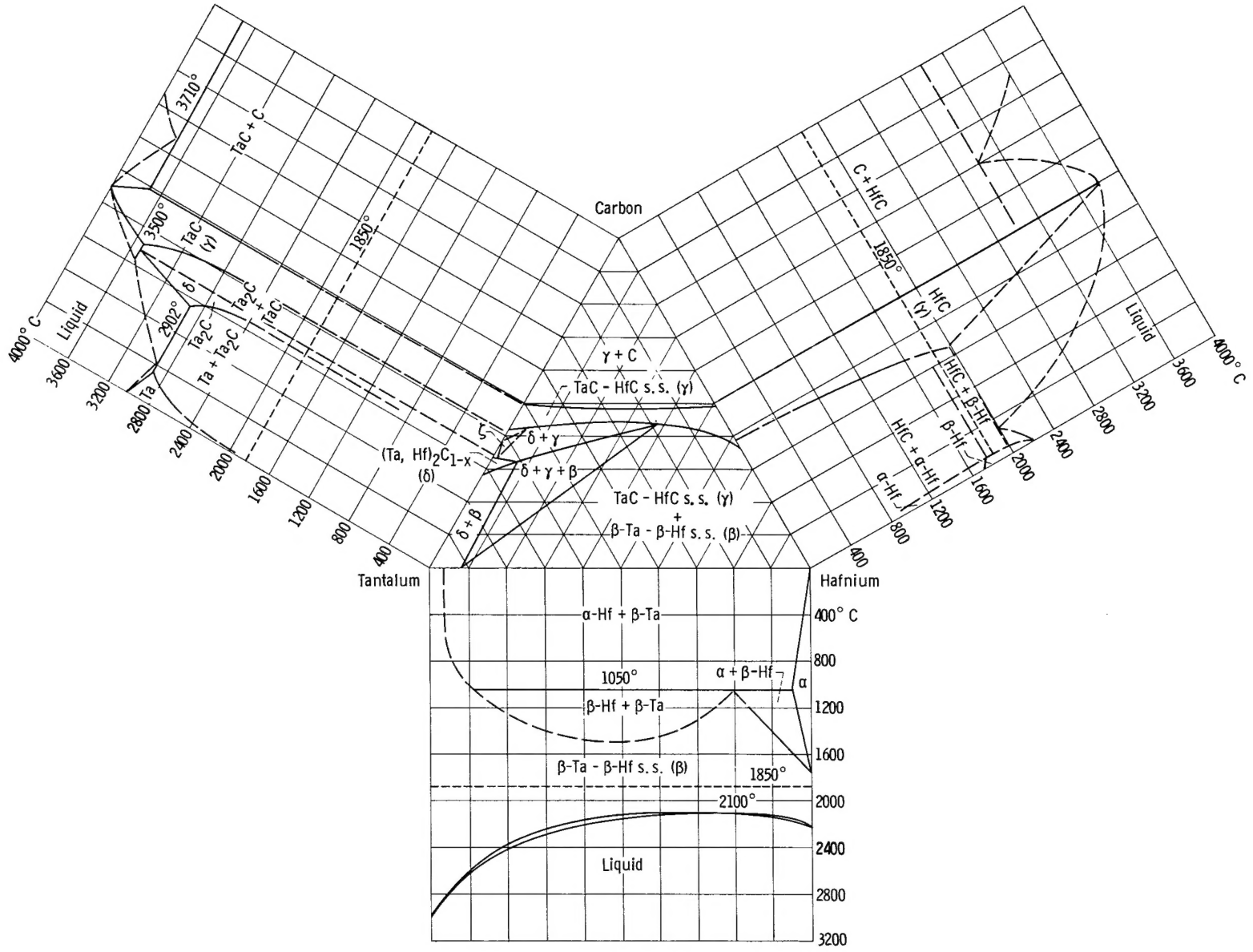


Figure 1. - Binary and ternary tantalum-hafnium-carbon systems given in literature. Ternary system is isothermal section at 1850° C.

EXPERIMENTAL PROCEDURE

Sample Preparation

The starting materials used in the preparation of the samples were tantalum hydride (TaH), hafnium-hydride (HfH_2), and spectrographic grade carbon. The analyses of the hydrides are given in table I.

Samples were prepared by three techniques: (1) sintering of cold-pressed mixtures, (2) hot pressing, and (3) sintering followed by arc melting. The cold-pressed and sintered specimens were prepared by blending the required amounts of the component powders followed by hydrostatically compacting (85 000 psi) at room temperature without a binder. These compacts, contained within thorium oxide crucibles, were set inside a tungsten susceptor and were slowly heated by induction in a vacuum of 5×10^{-6} to 9×10^{-6} torr to avoid rapid evolution of hydrogen. The specimens were brought up to 2050° C and held at that temperature for periods of from 1 to 30 hours until reaction was complete. The specimens were then cooled at a rate of 1000° C per minute to 900° C then more slowly to room temperature. The extent of reaction was determined by x-ray diffraction examination after each 1 to 5 hours of heating. Completion of reaction was assumed when the diffraction pattern showed no change. Several samples were checked for thorium content with an alpha counter. No thorium pickup was indicated.

Some compositions in the pseudobinary TaC-HfC region (about 50 atomic percent C) were hot pressed in graphite dies at 2400° to 2700° C at 3200 pounds per square inch for 1/2 to 1 hour.

Selected compositions were arc melted in argon using a nonconsumable tungsten electrode and a water-cooled copper hearth. The pickup of tungsten by the samples determined by chemical analysis was 0.01 to 0.1 weight percent.

All the hot-pressed and arc-melted samples were heat treated at 2050° C in vacuum for times up to 30 hours and cooled at a rate of 1000° C per minute to 900° C then more slowly to room temperature. This cooling rate is believed to be sufficiently rapid to retain the high-temperature phases except in hafnium-rich compositions where the precipitation of α-hafnium was observed. Even considerably faster cooling of composition 86 did not arrest the formation of α-hafnium. All sample compositions, along with phases present at room temperature, are given in table II.

It was also observed that sintered compositions 83, 85, 86, 87, 88 and 89, in the hafnium-rich corner of the diagram, showed large shrinkages, some deformation, and greatly increased densities. These observations suggest the presence of a liquid phase at 2050° C.

Measurements

Since it was expected that carbon loss would occur during preparation and heat treatment, all final products were analyzed for total and free carbon and the final composition calculated accordingly.

Temperature determinations were made with a disappearing-filament-type micro-optical pyrometer calibrated against a standard tungsten strip lamp (including the optical prism, which was part of the furnace). Temperature was determined with an accuracy of $\pm 30^{\circ}$ C.

A room-temperature x-ray diffraction study was done on powdered samples (taken from the center of the specimens) with nickel-filtered copper radiation using a diffractometer. In the case of metal-phase-rich compositions, which could not be easily ground, the patterns were taken from the interior cross section of the specimen. The lines on each pattern were indexed without difficulty because the structures of the phases involved were cubic or hexagonal. Lattice parameters were calculated on a 7094 computer by the least-squares method using the $1/\sin^2\theta$ extrapolation function. The standard deviation of all lattice parameters was ± 0.005 angstrom.

All arc-melted and some of the sintered compositions were examined by a metallographic technique. Etching was done with a mixture of hydrofluoric acid, nitric acid, and water. The composition of the etchant varied from one sample to another. When possible, the Knoop microhardness of the phases was determined with a 50-gram load. The hardness numbers reported are the average of 15 indentations. The standard deviation is ± 10 percent of the reported value.

RESULTS

X-ray Diffraction Study

The examination of the x-ray patterns revealed that no phases were present other than those appearing in the three binary systems. These are the face-centered-cubic $\gamma(Ta,Hf)C_{1-x}$, hexagonal $\delta(Ta,Hf)_2C_{1-x}$, hexagonal $\alpha(Hf,Ta)$, and body-centered-cubic $\beta(Ta,Hf)$ phases. Of course, the lattice parameters of these phases changed with the composition. This change was particularly large in the case of the TaC-HfC solid solution. The number and identity of the phases in each composition were determined as shown in figure 2. Each composition is identified by its sample number (composition). The same information is tabulated in table II, where, in addition to the number and identity of the phases, the lattice parameters are listed. Since x-ray diffraction patterns were taken at room temperature, the Ta-Hf body-centered-cubic solid solutions in the hafnium-rich corner were in some cases not observed, but instead body-centered-cubic tantalum and the hexagonal form of hafnium were detected. However, it is known from the binary Ta-Hf system that at 2050° C there is only one phase present, the $\beta(Ta,Hf)$ body-centered-cubic solid solution. In some instances when a phase was detected, but there were not enough diffraction lines for accurate determination of the lattice parameter, only its presence was indicated (table II). Since TaC-HfC solid solutions are of considerable practical interest because of their high melting points, their lattice parameters are shown as a function of composition in figure 3. The iso-lattice parameter lines are also drawn with the aid of data taken from references 3, 7, and 8.

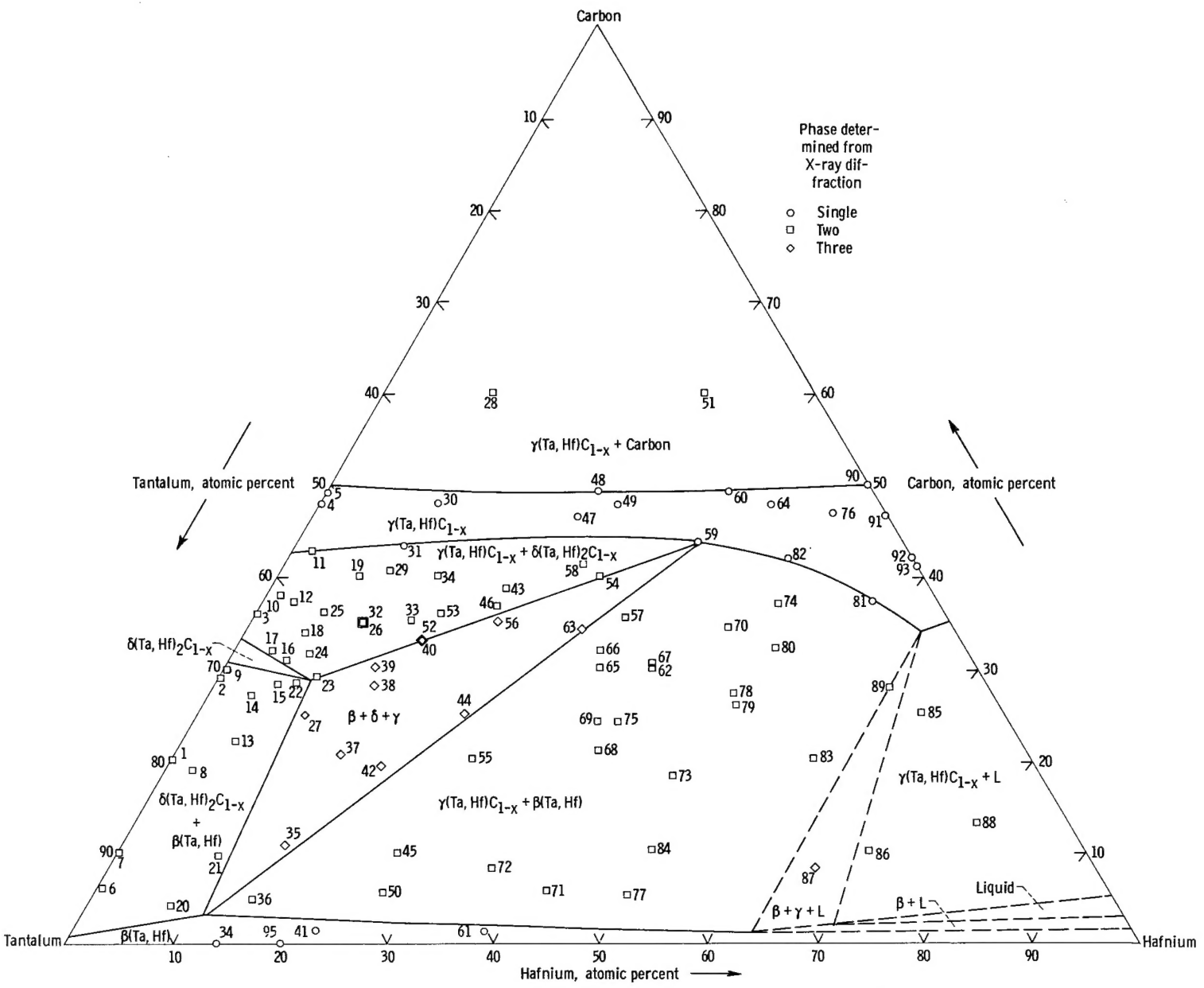


Figure 2. - Proposed isothermal cross section of tantalum-hafnium-carbon system at 2050° C.

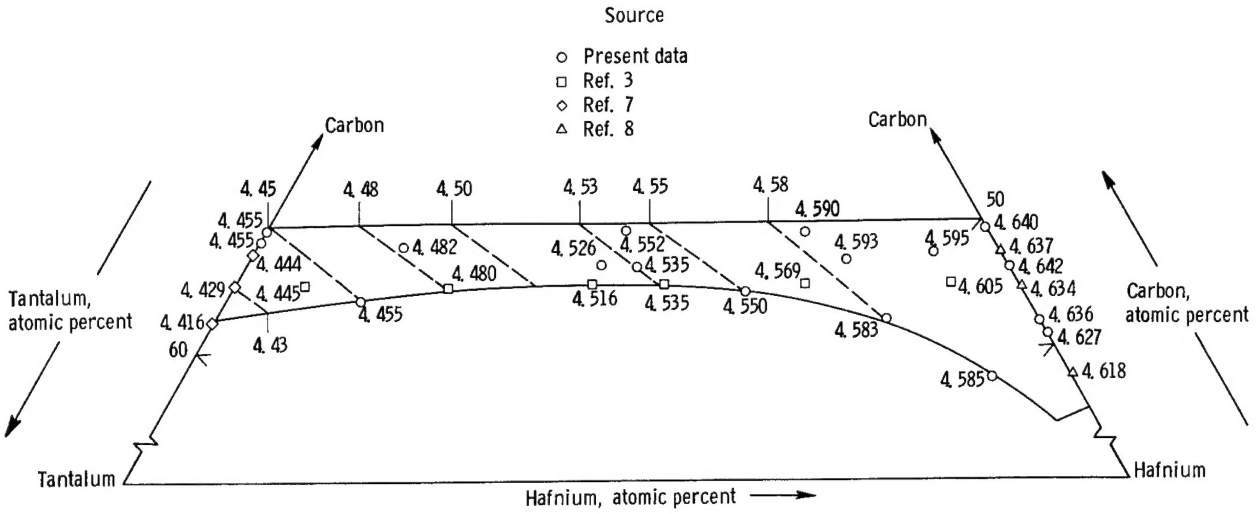


Figure 3. - Lattice parameters in face-centered-cubic (γ) single-phase region of tantalum-hafnium-carbon system.

Metallography

The sintered samples containing only small amounts of metallic phase were porous and could not be prepared for metallographic examination. The sintered samples, which contained a significant amount of metallic phase, and the samples which were arc melted, were mounted and polished with diamond compound.

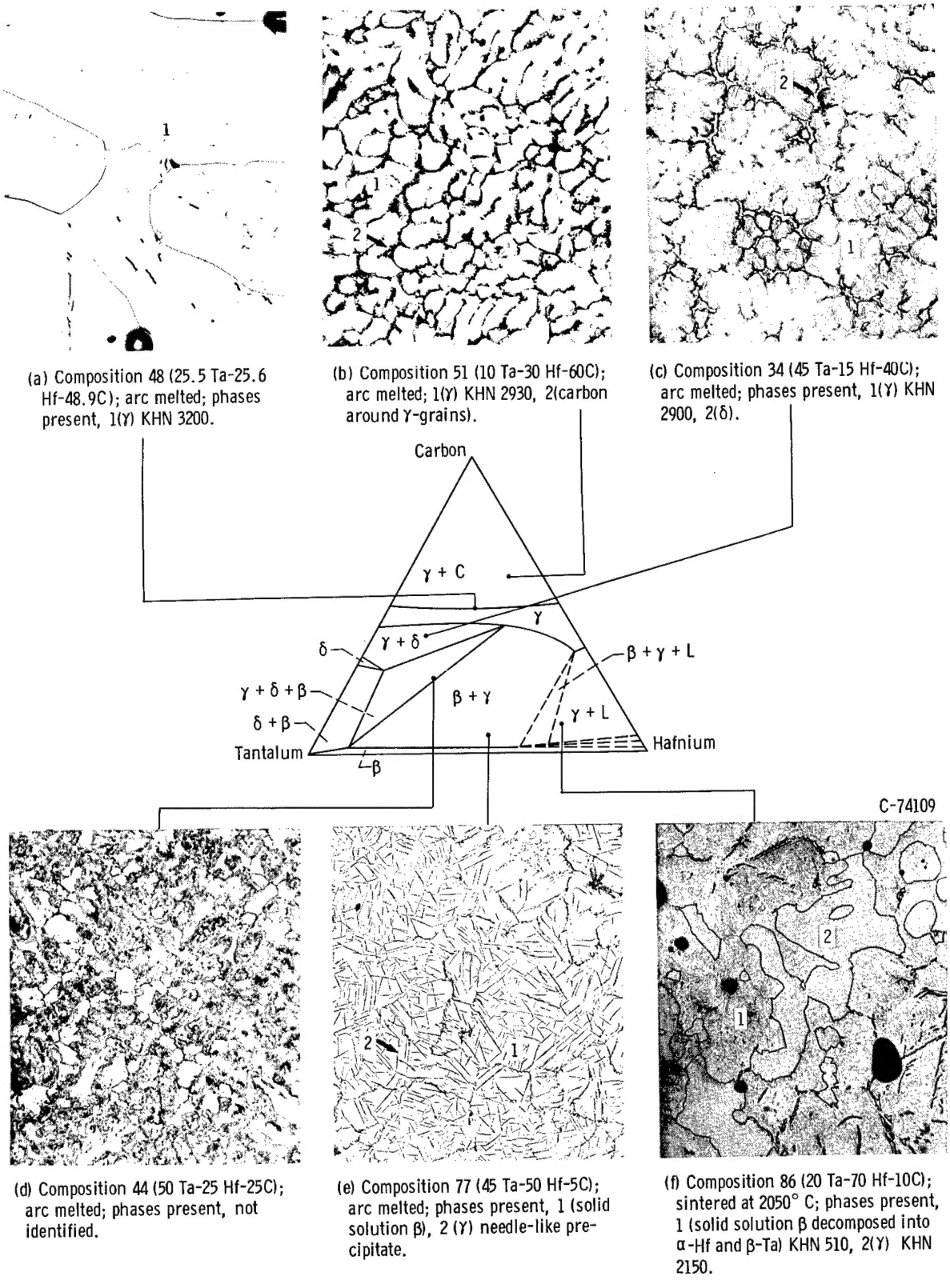
There is agreement between results obtained from metallography and x-ray investigation concerning the number of phases present in a given sample. Representative microstructures of sintered and arc-melted samples are shown in figure 4. The numbers in parentheses indicate the content of Ta, Hf, and C, respectively, in atomic percent. The phases are labeled and their microhardness (Knoop Hardness Number (KHN)) noted.

Figure 4(a), composition 48, is a typical microstructure of compositions in the region of the TaC-HfC pseudobinary system. With the exception of a few black areas, possibly due to porosity, impurities, or free carbon, only the γ-phase $(Ta,Hf)C_{1-x}$ (with nearly maximum carbon content) is present.

The structure shown in figure 4(b) is typical of the $\gamma(Ta,Hf)C_{1-x}$ + carbon phase field. Here completely carbon-saturated grains of γ-phase are found surrounded by carbon.

Composition 34, shown in figure 4(c), is typical of the $\gamma(Ta,Hf)C_{1-x}$ + $\delta(Ta,Hf)_2C_{1-x}$ phase field. The major component is the striated γ-phase. Similar striated structures have been observed in Ta-C binary compositions and reference 9 suggests these are the result of Ta_2C precipitation. The non-striated, intergranular component is the lower melting δ-phase.

Composition 44, within the three-phase area, yields a quite indiscernible microstructure, as shown in figure 4(d). The light areas are probably



(a) Composition 48 (25.5 Ta-25.6 Hf-48.9C); arc melted; phases present, 1(γ) KHN 3200.

(b) Composition 51 (10 Ta-30 Hf-60C); arc melted; 1(γ) KHN 2930, 2(carbon around γ-grains).

(c) Composition 34 (45 Ta-15 Hf-40C); arc melted; phases present, 1(γ) KHN 2900, 2(δ).

(d) Composition 44 (50 Ta-25 Hf-25C); arc melted; phases present, not identified.

(e) Composition 77 (45 Ta-50 Hf-5C); arc melted; phases present, 1 (solid solution β), 2 (γ) needle-like precipitate.

(f) Composition 86 (20 Ta-70 Hf-10C); sintered at 2050° C; phases present, 1 (solid solution β decomposed into α-Hf and β-Ta) KHN 510, 2(γ) KHN 2150.

Figure 4. - Typical microstructures in tantalum-hafnium-carbon system. X250. Reduced 45 percent in printing.

γ-phase $(Ta,Hf)C_{1-x}$. An x-ray diffraction pattern taken from the sample revealed the presence of three phases, $\gamma(Ta,Hf)C_{1-x}$, $\delta(Ta,Hf)_2C_{1-x}$, and $\beta(Ta,Hf)$.

The two-phase $\gamma(Ta,Hf)C_{1-x} + \beta(Ta,Hf)$ equilibrium area is represented in figure 4(e). The matrix is mostly β-phase, (Ta,Hf), with a needle-like precipitate of γ-phase, $(Ta,Hf)C_{1-x}$. Here one would expect to see an α-hafnium phase as a result of the decomposition of $\beta(Ta,Hf)$ solid solution (at 2050° C) on cooling. This was not the case, however, and the absence of α-hafnium was also verified by the x-ray diffraction method.

The photomicrograph of composition 86 is shown in figure 4(f). The metallic phase, which was liquid at 2050° C, yielded, on cooling, a mixture of α-Hf and β-Ta. The γ-phase, $(Ta,Hf)C_{1-x}$, forms large grains of irregular shape surrounded by the fine-textured α-Hf + β-Ta mixture. After heat treatment at 2050° C this specimen was deformed as a result of the presence of a liquid phase.

Microhardness

Microhardness information from references 10 to 12 as well as present data are shown in figure 5. The Knoop hardness values for the γ-phase, $(Ta,Hf)C_{1-x}$, vary from 1100 kilograms per square millimeter for carbon-deficient TaC to 3720 kilograms per square millimeter, when the hafnium content reaches about 11 atomic percent and the carbon is 48 atomic percent. Further increase in hafnium content, while approximately the same carbon content is maintained, causes a decrease in hardness. Completely carburized γ-phase compositions, in particular $TaC_{1.0}$, are significantly softer than carbon-deficient compositions. Figure 5 also shows that the hardness of the β-phase, (Ta,Hf), in the $\gamma + \beta$ field appears to increase with increasing hafnium content; however, the metallic matrix formed on cooling the composition in the $\gamma + L$ area is softer even though the hafnium content is greater than compositions in the $\gamma + \beta$ field. The γ-phase $(Ta,Hf)C_{1-x}$ in the $\gamma + L$ area is also softer than that in the $\gamma + \beta$ region.

DISCUSSION

On the basis of the previously described results, the phase boundaries shown in figure 2 were traced, and an isothermal section at 2050° C was constructed. The hafnium-rich corner of the diagram contains a liquid phase and the position of the phase boundaries were deduced primarily from the Ta-Hf and Hf-C binary systems and the observed deformation of compositions of high hafnium content due to liquid formation.

In general, the phase relations at 2050° C are similar to those at 1850° C except for the presence of the liquid phase. No phases other than those present in the binary systems were observed. The ζ-phase sometimes found in the binary Ta-C system and shown as a dotted area in the ternary system at 1850° C (fig. 1) was not detected at 2050° C. The solubility of carbon in the β-phase is about 2 to 3 atomic percent. The solubility of carbon in tantalum at 2050° C is given as about 2.2 atomic percent in reference 13. The transformation of the β-phase, (Ta,Hf) solid solution into β-Ta and α-Hf at the

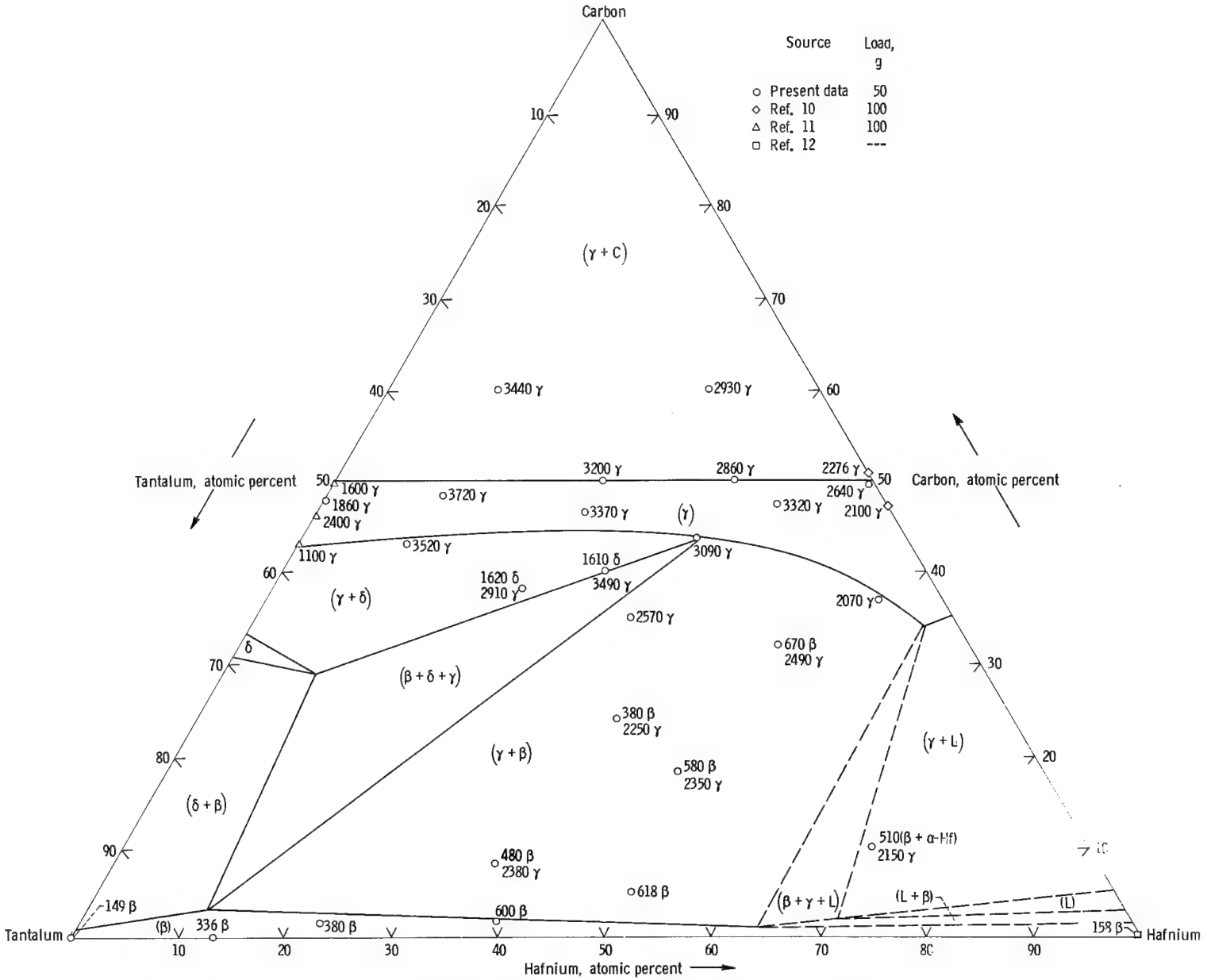


Figure 5. - Knoop hardness values of phases in tantalum-hafnium-carbon system.

cooling rate used in this study occurs only at high hafnium contents.

The $(Ta,Hf)_2C_{1-x}$ phase does not accept more than about 8 atomic percent hafnium at 2050° C. This occurs at the expense of tantalum and is accompanied by an increase in lattice parameter from a_o = 3.108 angstroms, c_o = 4.948 angstroms for Ta_2C to a_o = 3.120 angstroms and c_o = 4.970 angstroms for hafnium-saturated Ta_2C.

There is complete solid solubility between TaC and HfC, and the carbon content in the γ-phase, $(Ta,Hf)C_{1-x}$, may vary significantly. A hardness maximum appears to occur in the vicinity of 11 atomic percent hafnium, 41 atomic percent tantalum, and 48 atomic percent carbon (composition 30), which is near the compositions for which the highest melting point (ref. 1) and lowest vaporization rate (ref. 2) have been reported.

CONCLUSIONS

An isothermal cross section of the Ta-Hf-C system at 2050° C was constructed from x-ray diffraction, chemical analysis, metallographic, and microhardness data. The complete solid solubility between TaC and HfC was verified. The lattice parameters of $(Ta,Hf)C_{1-x}$ solutions were determined. No phases other than those appearing in the three binary systems were observed. A liquid phase was found to be present in a large portion of the hafnium-rich corner. There is an exchange of hafnium for tantalum in the $\delta(Ta,Hf)_2C_{1-x}$ phase up to about 8 atomic percent. The lattice parameters increase correspondingly to a_o = 3.120 angstroms and c_o = 4.970 angstroms.

The ζ-phase, sometimes found in the Ta-C system, was not observed in any of the compositions investigated at 2050° C. The limit of the $\gamma(Ta,Hf)C_{1-x}$ + $\delta(Ta,Hf)_2C_{1-x}$ area extends to about 38 atomic percent hafnium, which agrees with that at 1850° C. The solubility of carbon in the $\beta(Ta,Hf)$ solid solution is about 2 to 3 atomic percent.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, January 26, 1965.

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TABLE I. - CHEMICAL ANALYSIS OF

STARTING MATERIALS

Tantalum hydride	
Element	Weight percent
H	0.49
O	.072
N	.031
Si,W	0.005 - 0.05
Fe,Zr	0.01 - 0.10
Ti,Cu,Ni,V,Mn,Nb, Ca,Mg,Pb,Ba,K	<0.01
All other elements	All other elements not detected
Hafnium hydride	
H	1.1
Zr	1.6
O	.02
N	.003
Fe	.025
C	.015
B,U,Cd	<.001
Al,Mo,Si,W,Cr,Pb, Ni,Na,Co,Mg,Sn, V,Ca,Cu,Mn,Ti	0.001 - 0.01

TABLE II. - COMPOSITION, PREPARATION, AND ROOM TEMPERATURE X-RAY DIFFRACTION DATA

Sample number	Composition, atomic percent			Method of preparation	Number of phases detected by room temperature x-ray diffraction	Phases present by room-temperature x-ray diffraction and lattice parameters[a]					
	Tantalum	Hafnium	Carbon			β(Ta,Hf)	α(Hf,Ta)		δ(Ta,Hf)$_2$C$_{1-x}$		γ(Ta,Hf)C$_{1-x}$
						Body centered cubic	Hexagonal		Hexagonal		Face centered cubic
						a_o	a_o	c_o	a_o	c_o	a_o
1	80	0	20	Sintered	2	3.306	[b]N.D.	N.D.	3.103	4.938	N.D.
2	71	↓	29	Sintered	2	N.L.	↓	↓	3.103	4.948	N.D.
3	64		36	Arc-melted	2	N.D.			3.108	4.948	N.L.
4	52		48	Hot pressed	1	N.D.			N.D.	N.D.	4.455
5	51	↓	49	Sintered	1	N.D.	↓	↓	N.D.	N.D.	4.455
6	93.5	.5	6	↓	2	3.309			N.L.	N.L.	N.D.
7	89.8	.2	10			3.308			3.119	4.968	↓
8	78.5	2.7	18.8			N.L.			3.111	4.955	
9	70.1	.1	29.8			N.L.			3.109	4.942	
10	60.6	1.5	37.9			N.D.			3.106	4.942	4.414
11	55.6	1.7	42.7			N.D.			N.L.	N.L.	4.419
12	60	3	37			N.D.			3.110	4.949	4.451
13	73	5	22			3.309			3.116	4.959	N.D.
14	69	4	27			3.312			3.122	4.971	N.D.
15	65.9	5.9	28.2			N.L.			3.114	4.950	N.D.
16	64	5.2	30.8			N.D.			3.111	4.943	N.L.
17	64.5	3.4	32.1			↓			3.107	4.946	4.437
18	60.7	5.4	33.9						3.109	4.955	N.L.
19	52.5	7.5	40						N.L.	N.L.	4.418
20	87.9	7.8	4.3			3.316			3.125	4.971	N.D.
21	81.1	9.4	9.5			N.L.			3.137	5.003	N.D.
22	64.1	7.8	28.1			N.L.			3.115	4.954	N.D.
23	75	9.0	29.1			N.D.			3.114	4.942	N.L.
24	61.3	7.2	31.5			↓			3.101	4.940	N.L.
25	57.7	6.6	35.7						3.106	4.945	4.444
26	54.5	10.5	35	↓	↓				3.121	4.971	4.504
27	65	10	25		3	3.321			3.129	4.993	4.558
28	30	10	60	Arc melted	2	N.D.			N.D.	N.D.	4.489 + Carbon
29	49.3	10	40.7	Arc melted	2	↓			N.L.	N.L.	4.442
30	41	11	48	Hot pressed	1				N.D.	N.D.	4.482
31	46.7	9.9	43.4	Arc melted	1				N.D.	N.D.	4.455
32	55	10.3	34.7	Sintered	2				3.125	4.981	4.534
33	50	15	35	↓	2				3.120	4.963	4.500
34	45	15	40		2				3.112	4.954	4.504
35	74	15.2	10.8		3	3.319			N.L.	N.L.	4.556
36	80	15	5	Arc melted	2	3.338			N.D.	N.D.	N.L.
37	64	15.6	20.4	Sintered	3	3.324			N.L.	N.L.	4.547
38	57	15	28	↓	↓	N.L.			3.132	4.934	4.543
39	56	14	30			N.L.			3.125	4.935	4.553
40	50	17	33			N.L.			3.131	4.986	4.557
41	75.7	22.9	1.5	↓	1	3.352			N.D.	N.D.	N.D.
42	61	20	19		3	3.310			N.L.	N.L.	4.556
43	39.4	22	38.6	Arc melted	2	N.D.			N.L.	N.L.	4.471
44	50	25	25	Sintered	3	3.321			N.L.	N.L.	4.546
45	64	26	10	Sintered	2	3.346			N.D.	N.D.	4.585
46	41.5	21.5	37	Sintered	2	N.D.			3.121	4.955	4.510
47	28.5	25.1	46.4	Arc melted	1	↓			N.D.	N.D.	4.526
48	25.5	25.6	48.9	Hot pressed	1				↓	↓	4.552
49	24.7	27.7	47.6	Arc melted	1						4.535
50	67.4	26.7	5.9	Sintered	2	3.361	↓	↓			N.L.
51	10	30	60	Arc melted	2	N.D.	N.D.	N.D.	N.D.	N.D.	4.578 + Carbon
52	50.5	17.5	32	Sintered	3	N.L.	↓	↓	3.128	4.979	4.550
53	47	17	36	↓	2	N.D.			3.111	4.946	4.532
54	30	30	40		2	N.D.			3.127	4.931	4.527
55	52.5	27.5	20		2	3.321			N.D.	N.D.	4.552
56	42	23	35	↓	3	N.L.			3.120	4.979	4.544
57	29.8	34.7	35.5	Arc melted	2	N.D.			N.L.	N.L.	4.511
58	31	28	41	Hot pressed	2	↓			3.121	4.969	4.546
59	19.6	36.9	43.5	Arc melted	1				N.D.	N.D.	4.550
60	12.8	37.8	49.5	Hot pressed	1				N.D.	N.D.	4.590
61	59.2	39.4	1.4	Sintered	1	3.363				N.D.	N.D.
62	30	40	30	Sintered	2	N.D.				N.L.	4.585
63	34.5	31.5	34	Sintered	3	N.L.				N.L.	4.543
64	10	42.6	47.4	Arc melted	1	N.D.				N.D.	4.593
65	35	35	30	Sintered	2	3.324				N.D.	4.551
66	34	34	32	↓	↓	N.L.				↓	4.553
67	29	40	31			3.356					4.534
68	39.5	39.5	21			N.L.					4.595
69	38	38	24			3.373					4.594
70	20.4	45.1	34.5	Arc melted		N.L.					4.545
71	52.3	42	5.7	Sintered		3.367					N.L.
72	56	36	8	Sintered		3.371					4.590
73	34	48	18	Sintered		3.372					4.596
74	15	48	37	Arc melted		N.L.					4.556
75	36.5	39.5	24	Sintered	↓	3.364					4.538
76	5	48	47	Arc melted	1	N.D.					4.595
77	45	50	5	Sintered	2	3.403					4.611
78	24	49	27	↓		3.388					4.602
79	24.5	49.5	26			N.L.					4.602
80	17.5	50.5	32			N.L.					4.581
81	6	57	37		1	N.D.					4.583
82	11.5	46.5	42		1	N.D.	↓	↓			4.585
83	20	60	20		3	N.L.	N.L.	N.L.			4.598
84	40	50	10		2	3.363	N.D.	N.D.			4.602
85	7.5	67.5	25		3	N.L.	3.232	5.094			4.609
86	20	70	10		3	N.L.	3.187	5.064			4.595
87	26	66	8		3	3.370	3.196	5.096			4.602
88	8.5	78.5	13		2	N.D.	3.209	5.094			4.608
89	8.7	63.8	27.5		2	↓	3.222	5.123			4.626
90	0	50.4	49.6	Hot pressed	1		N.D.	N.D.			4.640
91	0	53.8	46.2	Sintered	↓		↓	↓			4.642
92	0	58	42	↓							4.636
93	0	59	41								4.627
94	86	14	0			3.329					N.D.
95	80	20	0	Arc melted		3.334					N.D.

[a]All lattice parameters are given in angstrom units, with a standard deviation of ±0.005.

[b]Phase was not detected by x-ray diffraction.

[c]Phase was detected, but because too few lines were present for accurate calculation of the lattice parameter, none is given.

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